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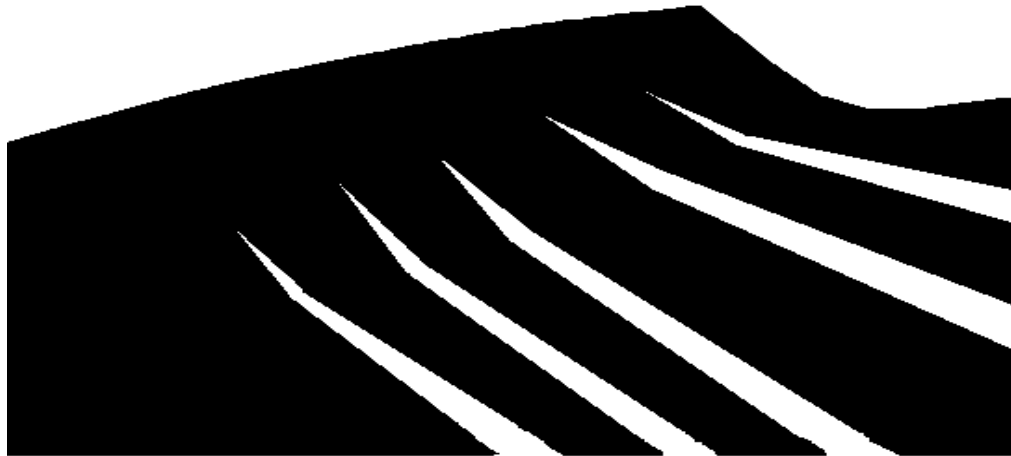
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LANL-CST-DP-68, R2

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## NTS FRACTURE CORE EXPERIMENTS

### ***LOS ALAMOS QUALITY PROGRAM***



#### APPROVAL FOR RELEASE

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**Los Alamos**

Yucca Mountain Site

Characterization Project

## HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	04/03/89	N/A	Initial procedure.
R1	02/13/96	All	Complete rewrite to update and renew processes. Revision 0 of this procedure was previously identified as LANL-INC-DP-68.
R2	03/21/97	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

**Los Alamos**Yucca Mountain Site  
Characterization Project

## **NTS FRACTURE CORE EXPERIMENTS**

### **1.0 PURPOSE**

This procedure is designed to study transport mechanisms within saturated fracture cores (i.e., cores saturated with groundwater) permitting the close simulation of an actual field experiment.

### **2.0 SCOPE**

This procedure applies to the final fabrication, assembly, preparation and sampling of an NTS fracture core experiment.

### **3.0 REFERENCES**

LANL-CST-DP-60, Preparation of NTS Samples for LANL YMP Solid Core Experiments.  
LANL-YMP-QP-02.7, Personnel Training.  
LANL-YMP-QP-03.5, Documenting Scientific Investigations.  
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards.  
CST-SOP-037, Rules for Handling Radioactive Material.  
LANL-YMP-QP-08.1, Identification and Control of Sample.

### **4.0 DEFINITIONS**

#### **4.1 Tracer Spike**

Solution containing the substance(s) to be injected into the fracture core for the study of transport behavior.

#### **4.2 Tracer Solution**

Solution containing the substance(s) to be loaded onto the fracture core for the study of transport behavior.

#### **4.3 Elute**

The solution resulting from the elution process.

#### **4.4 Collection Time**

Time interval between collection of elute samples.

#### 4.5 Flow Rate

The amount of liquid eluted through the fracture core per unit of time.

#### 4.6 $C_0(t)$

Tracer concentration in initial tracer solution or spike at time  $t$ .

#### 4.7 $C_i(t)$

Tracer concentration in solution I at time  $t$ .

### 5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- Principle Investigator (PI)
- Users of this Detailed Procedure (DP)

### 6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, subsection 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

#### 6.1 Principle

The presence of fractures in the geohydrologic system makes modeling of contaminant transport difficult. The reasons are twofold. The properties of the medium become heterogeneous and the interaction of radionuclides with fracture minerals are also heterogeneous. This procedure permits the simulation of an actual field experiment using fracture cores in a controlled laboratory environment.

## 6.2 Equipment and Hardware/Software

- Plexiglas: tubing, rod, flat stock
- Tygon tubing
- Low pressure chromatography: fittings and tubing
- O-rings
- Rock saw, wire saw, or slab saw
- Sealing compound: epoxy, silastic
- Machine shop tools: lathe, milling machine, drill press, assorted hand tools
- Solution pump, peristaltic pump, or syringe pump
- Fixtures for holding final assembly
- Calibrated balance
- Fraction Collector
- Oven
- Injection valve (if tracer spike is to be used)
- Sample Loop (if tracer spike is to be used)

### 6.2.1 Equipment Malfunctions

The equipment needed to conduct this experiment must be clean and in good operating order. Do not exceed the capacity of the Sage syringe pumps to handle more than 35 pounds of pressure. The computerized syringe pumps are susceptible to surges, spikes and general powerfailure such that they instantly turn themselves off, in this event the pumps must be turned back on.

### 6.2.2 Safety Considerations

Ensure Compliance with CST Division Environmental Safety and Health Operational Policy Statement.

### 6.2.3 Special Handling

Tracer solutions for these experiments are often radioactive and should be handled in accordance with Rules for Handling Radioactive Material at TA-48 (CST-SOP-037). Injection of radionuclides often leaves residue sorped to minerals of the rock column. The column after injection should be handled as potentially contaminated and disposed of a slow level waste.

## 6.3 Preparatory Verification

There are no critical setup parameters, prerequisites, or mandatory verification points.

### 6.3.1 Hold Points

There are no hold points for this procedure.

### 6.3.2 Calibration

The balance(s) used in this procedure must be calibrated according to QP-12.3. When data are collected from a balance, the unique identifier number of that balance must be recorded in the user's laboratory notebook along with the data collected. Micrometers and calipers are not calibrated because they are used as a convenience. Ruler accuracy is adequate.

### 6.3.3 Environmental Conditions

Column elutants must be collected within a highly humidified sealed fraction collector. The fraction collector must be sealed for two reasons: First, in case of spillage the fraction collector is used as containment. Second, evaporation of column elutants causes inaccuracy in measuring radioactivity based on weight; therefore, keeping the humidity of the fraction collector high, reduces evaporation of the elutants. Humidity is kept artificially high by filling large mouth containers with water within the sealed fraction collector.

## 6.4 Control of Samples

The unique identifier of the fracture core will be carried from DP-60. If more than one fracture core has been prepared from the same NTS sample an additional alphanumeric character will be added to the identifier in order to maintain uniqueness. Sample identification and control are governed by QP-08.1.

## 6.5 Implementing Procedure (See Attachments 1 and 2).

### 6.5.1 Preparation of Fractured Rock Core for Column Experimentation

The two ends of the fracture core must be cut with a rock saw to produce straight edges. Retain the resulting surplus material for utilization in mineral and chemical analysis of the fracture and for porosity measurements. To prevent slippage and/or damage of the fracture, a hoseclamp must be used to secure the core.

Measure and record the dimensions for the length and width of the core and of the fracture, particularly if the dimensions of the fracture differ from that of the core.

#### 6.5.2 Preparation of Column Endcaps

Round endcaps are made for  $\frac{1}{2}$  to 1" flat Plexiglas. The diameter of the endcaps are made slightly larger than the core by approximately  $\frac{1}{8}$ ", in order to produce an overlip. The endcaps are then cored within the overlip, at a depth of  $\frac{1}{16}$ " to  $\frac{1}{8}$ ", the exact diameter as the rock core. The endcaps are then fitted onto the core and a trace of the fracture is drawn on the caps using a marking pen. Troughs  $\frac{1}{16}$ " deep are cut using a mill over the designated area. Caution must be applied in order not to cut into the overlip. Two areas measured at a distance of  $\frac{1}{8}$ " outside both sides of the trough and  $\frac{1}{8}$ " from the cored overlip are milled to the same depth as the troughs. (The two milled sections should resemble opposing D's).

Two  $\frac{1}{6}$ " holes are drilled through the troughs of each endcap. The holes need to be evenly aligned and centered to the troughs. The top of each hole is then redrilled to a depth of  $\frac{1}{4}$ " and tapped for a  $\frac{1}{4}$  x 28 thread.

#### 6.5.3 Attaching Endcaps to Fractured Core

The endcaps are secured to the fracture core by placing a thin coating of silastic within the overlip and the milled D-sections. The endcaps are then slowly and carefully fitted to the fractured rock core making sure to get no silastic within the trough or the fracture and making sure to align the trough with the fracture. Allow silastic to cure completely before proceeding.

Silastic is spread over all exposures of the rock core, including the fracture; however, a light touch, with caution, is required to prevent pushing silastic into the fracture. Allow silastic to cure completely before proceeding.

#### 6.5.4 Attaching Donut Spacer to Bottom of Column

A tube of transparent leucite, with a diameter greater than that of the endcaps is selected and cut such that the length will stretch to the middle of both endcaps. A piece of Plexiglas, similar in thickness to the endcaps, is then cut in donut form, so that it has an outer diameter equal to the inner diameter of the leucite tube and an inner diameter equal to that of the endcaps. The resulting donut is then pressed and sealed with silastic to one of the endcaps and then pressed and sealed with silastic to one end of the leucite tube. This end is then referred to as the bottom. Allow silastic to cure completely before proceeding.

#### 6.5.5 Encapsulation

Encapsulation is generally performed by using a non-shrinking epoxy. Care must be used to ensure correct mixing ratios and adequate stirring time. A cheaper substitute for epoxy, parafilm wax, is being studied and may turn out to be more viable product for encapsulation. To begin encapsulation, the fracture core is supported by a beaker or other container sufficient to keep the bottom of the core suspended. Epoxy/melted parafilm is then poured through the gap, at the top of the fracture core, between the leucite tubing and the silastic coated rock core. The epoxy/melted parafilm is poured until the leucite tube is filled. The final step in this process is following the requirements for correct curing of the epoxy. Generally this is to leave alone while curing in a vacuum hood. For setting of the parafilm, leave alone until set after cooling.

The dry weight of the fracture core is recorded after placing the fracture core into a vacuum over for 10 days at a temperature ranging from 50 to 70°C.

#### 6.5.6 Flushing Nitrogen from Column

In order to accelerate the saturation process, atmospheric nitrogen, which is insoluble in water, must be flushed from the column. One arbitrarily chosen inlet/outlet port of the fracture core is fitted with a plastic ¼ x 28 specialized bolt. The bolt must have a hole through the center to allow unrestricted flow of gas or liquid. The second port, which is on the same endcap with the specialized bolt, is sealed using a solid plastic ¼ x 28 bolt. A tube is fitted over the specialized bolt and connected to a source of CO<sub>2</sub> gas which is then opened and allowed to flush through the column for 30 minutes at a pressure ranging from 10 to 20 pounds. After 30 minutes has elapsed, while the gas is still flowing, the endcap which is outflowing the gas is sealed with plastic bolts. Because CO<sub>2</sub> gas is heavier than nitrogen, the fracture core is turned so that the sealed end is down. At this stage the gas is turned off and the two bolts, which includes the specialized bolt, are removed and then resealed with two fingers. Keeping the two fingers in place, the fracture core is inverted and quickly submerged into a beaker of well water, specified by the PI.

#### 6.5.7 Saturation by Vacuum

The beaker containing the submerged fracture core is placed in a vacuum system. The beginning vacuum pressure should be of moderate intensity (<15 inches Hg), so as not to displace the minerals coating the fracture walls. Generally after two weeks, a higher vacuum pressure (+30 inches Hg) can be used. Saturation is achieved when all air bubbles cease to be released or visibly seen within the endcap. This saturation time ranges from four to twelve weeks.



#### 6.5.8 Establish Groundwater Flow with the Pump

After saturation is completed, the fracture core is removed from the vacuum system. A pump is set up to flow well water through the column. All water used in the pump must be degassed to prevent regassing of the column. While the pump is flowing well water, insert a specialized plastic bolt, connected with a tube to the pump, into one of the ports. Seal off the adjacent port with a solid plastic bolt. The fracture core can now be removed from the beaker and placed onto a ring-stand. Position the fracture core such that the inlet is at the bottom flowing upward. Seal off one of the outflow ports with a plastic bolt and insert a specialized bolt with attached tubing to the remaining outflow port.

**NOTE:** *Use O-rings in all the ports.*

Establish a flow rate through the fracture. The flow rate is determined by measuring the amount of water collected with time.

#### 6.5.9 Conducting Experiment

6.5.9.1 Prepare the tracer solution or spike to be used.

6.5.9.2 Weigh, record and place the vials/tubes to be used for elute collection into the fraction collector.

6.5.9.3 If a tracer solution is used, place the traced solution into the pump. If a tracer spike is used, load and spike into the injection valve. Tracers are pumped through the bottom of the fracture core to the top to help reduce any induced or trapped air and to help disperse the tracer through the core.

6.5.9.4 Turn the collector on. Start the pump, and if a tracer spike is used, inject the spike. Record the Julian time. Place an aliquot of the tracer solution or spike into a counting container (the tracer concentration of this aliquot will be used to determine  $C_0$  at an arbitrary time  $t$ ). Promptly cap the vials used for elute collection after collection is attained.

6.5.9.5 Determine the amount of tracer in the initial solution and in the elutants collected using appropriate analytical measures.

6.5.9.6 Ensure that the following data are recorded in the Logbook/Binder Entries

- a) Unique identifier of the sample.
- b) The dimensions of the fracture core.
- c) Dry and saturated weights of the fracture core.

- d) The unique identifier of the water used.
- e) The flow through the fracture core.
- f) Tracer solution or spike to be used and analytical method preparation (e.g., the experimental steps used for preparation, notebook number and pages used specifying these steps, or a detailed procedure number).
- g) Analytical technique used to analyze tracer and reference to the detailed procedure for this technique.
- h) Collection time to be used on fraction collector.
- i) The starting time of elutant collection and amount of initial tracer solution or spike to be analyzed, specifying the units of volume or weight used for measuring amount.

## 6.6 Data Acquisition and Reduction

The tare and the final weight of elute vials to be used for flow rate determination are recorded. These data are later reduced by subtracting the tare weights from final weights of the collection vials and dividing by the elapsed time in order to obtain the flow rate through the fracture core. The relative concentration of the tracer,  $C_i(t)/C_0(t)$ , will be calculated for each elute collected.

## 6.7 Potential Sources of Error and Uncertainty

- 6.7.1 Evaporation of the tracer in the collection vials can cause errors in the results. Consequently, keep the collection vials capped after elute collection.
- 6.7.2 The most common source of error that results in data rejection is leakage. Leakage can be caused by loose fittings. Leakage is detected by periodic visual inspection. If leakage is detected, record this problem in the laboratory notebook and reject the data.

## 7.0 RECORDS

Records resulting from the proper execution of this DP are entries in the laboratory notebooks and electronic media on which data are stored. Generation of these records will follow QP-03.5.

## 8.0 ACCEPTANCE CRITERIA

Proper recording of the data specified in subsection 6.5.4 constitutes the acceptance criteria for this DP.

## **9.0 TRAINING REQUIREMENTS**

Formal training is required for this DP. Training is documented in accordance with QP-02.7.

## **10. ATTACHMENTS**

Attachment 1: Figure 1: Flow Diagram

Attachment 2: Figure 2: Cross Section

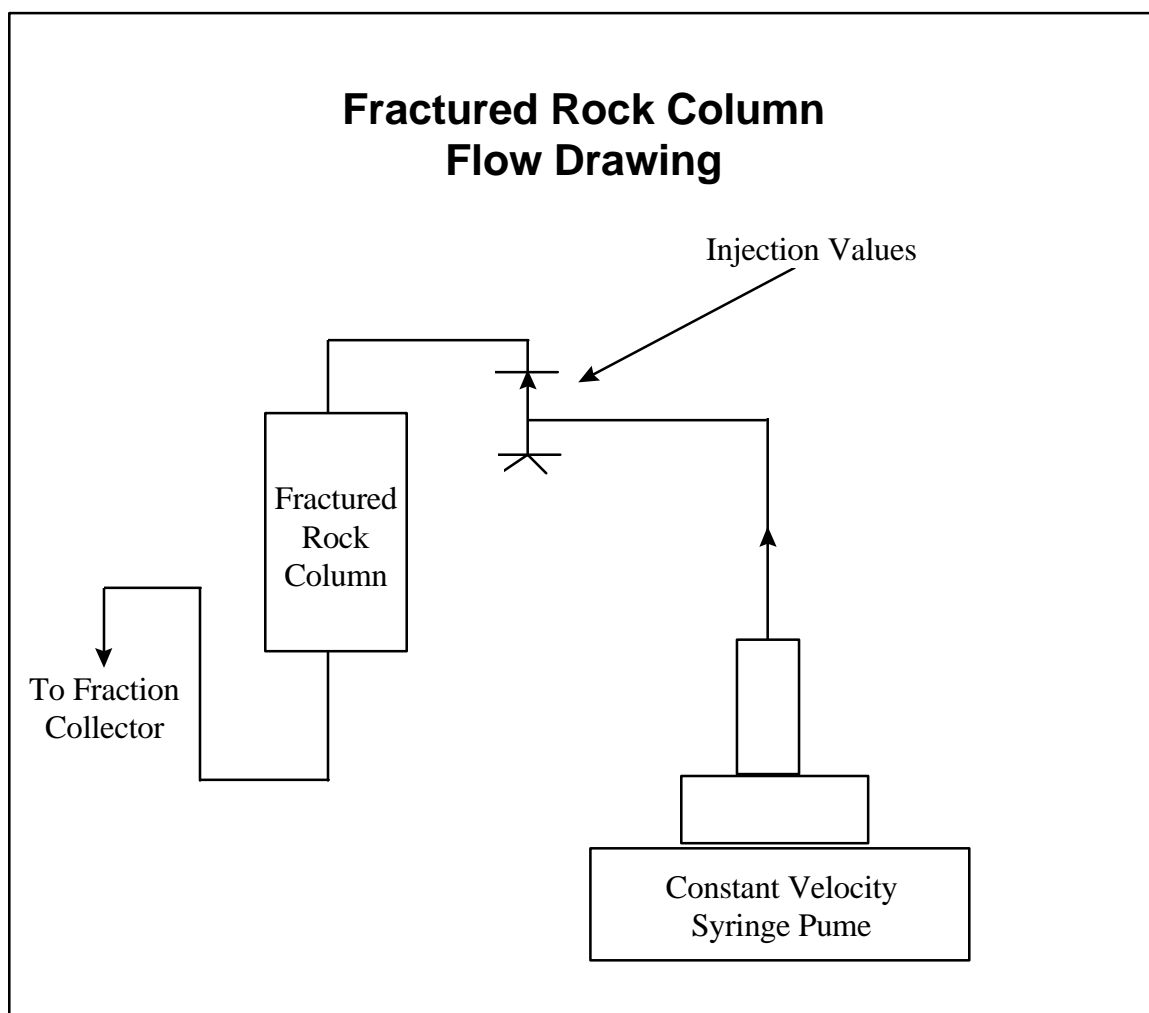


Figure 1

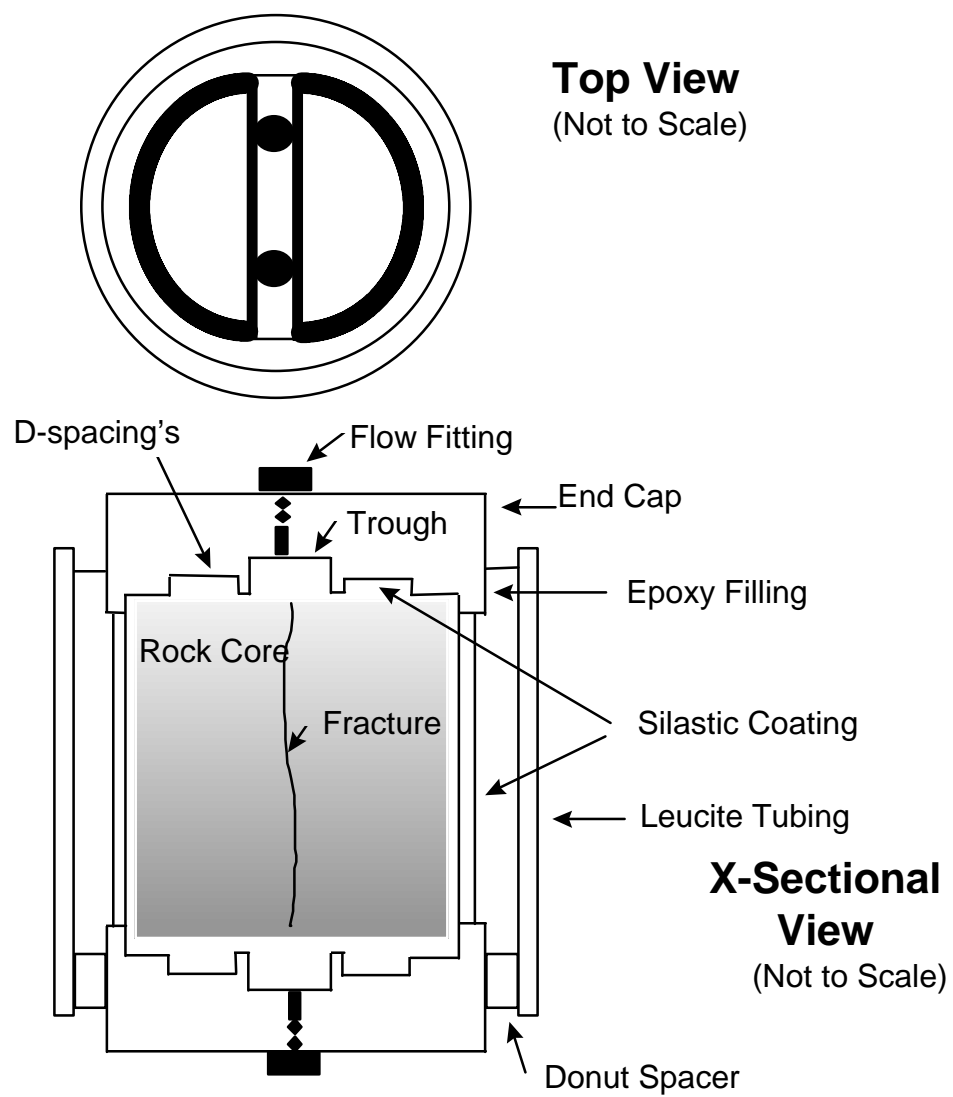


Figure 2